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(54) ELECTRICALLY CONDUCTIVE TIN OXIDE FINE POWDER AND ITS PRODUCTION

(57)Abstract:

PURPOSE: To obtain white electrically conductive fine powder excellent in electric conductivity by reacting a solution of a soluble tin compound with an acid or an alkali in the presence of a soluble phosphorus compound and burning the resultant phosphorus-containing tin hydroxide precipitate.

CONSTITUTION: A solution of a soluble tin compound is made to react with a solution of a soluble phosphorus compound and an aqueous solution of an acid or an alkali at 30-100°C or the solution of the soluble tin compound is allowed to react with the aqueous solution of the acid or alkali to provide a hydrous tin hydroxide precipitate. The soluble phosphorus compound is then added to the resultant hydrous tin hydroxide precipitate. Thereby, a phosphorus- containing tin hydroxide precipitate is obtained, then separated by filtration and washed to regulate the pH to 2-4. The regulated precipitate is subsequently burned at 800-1300°C for 1-5hr and pulverized to afford the objective electrically conductive fine powder, consisting essentially of SnO<sub>2</sub>, containing phosphorus in an amount expressed in terms of  $2.7 \times 10^{-2}$  to  $1.4 \times 10^{-1}$  atomic ratio (P/Sn) and having  $\geq 10 \text{ m}^2/\text{g}$  specific surface area and  $\leq 500 \Omega \text{ cm}$  powder resistance value.

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] The invention in this application relates to the conductive outstanding white conductivity powder and its conductive manufacture approach. The white conductivity powder of the invention in this application is still more useful as a conductive base material for electrophotography photo conductors as fillers, such as a coating aiming at electrification prevention, ink, plastics, rubber, and fiber, as electro-conductivity applying agents, such as electrophotography sensitized paper and electrostatic recording paper.

[0002]

[Description of the Prior Art] Although carbon black is known as conductive powder for many years, since there are many faults -- that the distribution to a vehicle with black color contains a bad carcinogen etc. receives various constraint on the occasion of use -- the titanium-dioxide powder which has the enveloping layer of the tin oxide which doped the tin-oxide powder which doped antimony, and antimony is developed, and this thing is used in recent years. The method of the conductive powder which does not use antimony being called for and replacing it with antimony from the field of safeties, such as the toxicity of antimony, recently, although antimony content tin-oxide powder is excellent in respect of conductivity, and making the tin oxide dope metals, such as germanium, Lynn, a lithium, and zinc, is proposed.

[0003] As the manufacture approach of the tin-oxide powder which does not use antimony For example, add the liquid which dissolved germanium, P, Li, and Zn in the tin chloride solution to \*\* alkali water solution, and it is made to generate precipitation. By holding the water solution of the approach (JP,2-32213,B) of calcinating this precipitation at 350-700 degrees C, and \*\* tin compound, under pH condition of 8-12, and hydrolyzing the tin compound in liquid gradually The sol containing the colloidal particle of a metallic oxide and/or a water oxide is made to generate. After collecting colloidal particles from this sol, the water solution of phosphorus compounds is infiltrated into said colloidal particle, and the method (JP,63-11519,A) of drying and calcinating this particle after an appropriate time etc. is learned.

[0004]

[Problem(s) to be Solved by the Invention] While examining many things in order to obtain the white powder which was excellent in conductivity without this invention person's etc. using antimony, paying attention to above-mentioned JP,2-32213,B, the approach of changing to especially antimony and making Lynn doping was examined. Consequently, to the tin-oxide powder which doped Lynn, knowledge that there are not few troubles which should still be solved -- \*\* passage-of-time stability further to be calcinated by reducing atmosphere is not enough to improve \*\* conductivity which is not enough in respect of \*\* conductivity -- was acquired.

[0005]

[Means for Solving the Problem] In order that this invention person etc. may solve the aforementioned problem, as a result of inquiring wholeheartedly, when making Lynn dissolve and giving high conductivity during \*\* tin-oxide crystal If narrow criticality nature is in the amount of dissolution of Lynn compared with the case where antimony is made to dissolve and it deviates from the range of  $2.7 \times 10^{-2}$  to  $1.4 \times 10^{-1}$  by the P/Sn atomic ratio, conductivity will fall rapidly and desired conductivity will not be acquired, \*\* A conductive predetermined thing is not obtained below 700 degrees C, but it is necessary to calcinate the temperature at the time of calcinating, after processing phosphorus compounds to the tin oxide at the temperature of 800 degrees C or more, \*\* When making Lynn dope, a header and the invention in this application were completed for sintering of a particle not arising, even if it calcinates at the temperature of 800 degrees C or more unlike the case where germanium, a lithium, and zinc are made to dope etc.

[0006] Namely, the invention in this application uses the tin oxide as a principal component, and Lynn is included by the P/Sn atomic ratio at a rate of  $2.7 \times 10^{-2}$  to  $1.4 \times 10^{-1}$ . It is the conductive impalpable powder characterized by being more than specific surface area of  $10 \text{ m}^2 / \text{g}$ , and being 500ohms below of fine-particles resistance cm. Moreover, [ whether a fusibility tin compound solution is made to react to the bottom of existence of (1) fusibility phosphorus-compounds solution with an acid or alkali, and ] Or it is made the water tin-oxide precipitate which is made to react with (2) acids or alkali, and is generated whether addition processing of the fusibility phosphorus compounds is carried out. It is the manufacture approach of the conductive impalpable powder characterized by obtaining the Lynn content water tin-oxide precipitate, classifying the precipitate obtained after an appropriate time, and calcinating and grinding at the temperature of 800-1300 degrees C.

[0007] It is the powder which the conductive impalpable powder of the invention in this application used the tin oxide as the principal component, and dissolved Lynn, and specific surface area is as follows [ more than  $10 \text{ m}^2 / \text{g}$  (based on a simple BET adsorption method), and 500ohms of fine-particles resistance cm ].  $5.0 \times 10^{-2}$  to  $9.0 \times 10^{-2}$  comes out comparatively desirably, and it contains. more -- a detail -- Lynn -- a P/Sn atomic ratio --  $2.7 \times 10^{-2}$  to  $1.4 \times 10^{-1}$  -- It has the presentation from which the remainder consists of the tin oxide substantially, and is the conductive impalpable powder which uses the tin oxide below 100-ohmcm as a principal component still more desirably below 200-ohmcm desirably more than  $15 \text{ m}^2 / \text{g}$ , and 500ohms below of fine-particles resistance cm more than specific surface area of  $10 \text{ m}^2 / \text{g}$ . there are few amounts of Lynn than the above-mentioned range -- many [ even if alike too much ] -- desired conductivity is hard to be acquired even if alike too much.

[0008] Although there is the approach of versatility [ make / the Lynn content water tin-oxide precipitate / generate ] in the manufacture approach of the conductive impalpable powder of the invention in this application (1) fusibility tin compound solution under existence of a fusibility phosphorus-compounds solution For example, an acid Or the method of settling the hydrated compound of the tin oxide with which it is made to react with alkali and phosphorus compounds are sticking to the surroundings of it, (2) The method of making a fusibility tin compound solution react with an acid or alkali, making water tin-oxide precipitate generate, carrying out addition processing of the fusibility phosphorus compounds after that, and making the Lynn content water tin-oxide precipitate generate etc. is mentioned. In the approach of the above (1) In addition, a fusibility tin compound solution, a fusibility phosphorus-compounds solution, and an acid The reaction with alkali Or for example, (a) fusibility tin compound solution, The approach of adding an acid or alkali and neutralizing in the mixed solution of the (b) fusibility tin compound solution and the fusibility phosphorus-compounds solution which it adds [ solution ]-like in parallel and make a fusibility phosphorus-compounds solution and an acid, or an alkali water solution react into hot water, is employable. neutralization with an acid or alkali -- the bottom of heating -- or it is desirable to carry out in hot water and 30-100 degrees C is preferably performed at 50-90 degrees C.

[0009] In the invention-in-this-application approach, although various things can be used as a tin compound, a stannic chloride, a stannous chloride, a stannic-acid potassium, stannic-acid sodium, etc. are mentioned, for example. Moreover, as phosphorus compounds, phosphorus-trichloride, orthophosphoric acid, dibasic-sodium-phosphate, phosphoric-acid 3 sodium, phosphate water \*\* ammonium, phosphorous acid, and phosphorous acid 2 hydrogen sodium, phosphorous acid 3 sodium, a phosphorus pentachloride, etc. are mentioned, for example, and a kind of these or two sorts or more of compounds can be used. It is good to use the solution which dissolved a tin compound and phosphorus compounds in a kind of alcohol, a hydrochloric-acid water solution, and the acetones, or two sorts or more of solutions or mixed liquor, respectively as a fusibility tin compound solution and a fusibility phosphorus-compounds solution. As an alkali water solution used as a neutralizer, a hydroxide, a carbonate, ammonia, etc. of alkali metal, such as a sodium hydroxide, a potassium hydroxide, a sodium carbonate, and potassium carbonate, are mentioned.

[0010] Next, in the invention-in-this-application approach, a neutralization product is classified and it calcinates at the temperature of 800-1300 degrees C. Judgment is usually filtered and is performed by washing if needed. In this case, it is desirable to filter a resultant, after adjusting pH of the reaction mixture after neutralization termination to 2-4 desirably five or less. Since washing is insufficient and alkali metal sticks to this resultant, and it will become the cause of reducing conductivity if it remains, it is necessary to perform sufficient washing, to use the hydroxide and carbonate of alkali metal as a neutralizer so that alkali metal may not remain.

[0011] After drying if needed after that, 800-1300 degrees C of 850-1250 degrees C of resultants classified and

acquired are desirably calcinated at the temperature of 900-1200 degrees C still more desirably. Although baking can be performed also in which [ of an oxidizing atmosphere, reducing atmosphere, and an inert gas ambient atmosphere ] ambient atmosphere, it is advantageous to carry out in air. Although it needed to calcinate at the temperature of 700 degrees C or less with the conventional method, it is the big description especially in the invention in this application that it can calcinate in air, without causing substantially big-and-rough-izing and sintering of the particle of a calcinated object, and it can give easily 700 degrees C or more of sufficient conductivity in high temperature 800 degrees C or more. In addition, although firing time changes with an equipment format, throughput, etc. and cannot generally be specified, 1 - 2 hours is preferably suitable for it for 1 to 5 hours. Grinding processing is performed according to the conventional method after baking, after this, if needed, pH of a grinding object can be adjusted or an impurity can also be removed.

[0012]

[Example]

Concurrency addition of the solution and sodium-hydroxide water solution which dissolved 500g ( $\text{SnCl}_4$  and  $5\text{H}_2\text{O}$ ) of stannic chlorides and 16.1g ( $\text{PCl}_3$ ) of phosphorus trichlorides in 500ml of 3-N hydrochloric acids was carried out over for 20 minutes, maintaining pH of a system to 7.0-7.5, and precipitate was made to generate in 5l. of water of 150 degrees C of examples. Next, after adding the hydrochloric acid and adjusting pH of a system to 3.0, this precipitate was filtered, and it washed until the specific resistance of filtrate was set to 10000-ohmcm after that. After drying the obtained cake at 110 degrees C for 12 hours, it calcinated at 1000 degrees C with the electric furnace for 1 hour, the PARUPE riser ground, and the conductive impalpable powder (sample A) made into the purpose of specific surface area of  $34\text{m}^2/\text{g}$  was obtained.

[0013] In example 2 example 1, except using 13.4g ( $\text{PCl}_3$ ) of phosphorus trichlorides, it processed similarly and the conductive impalpable powder (sample B) made into the purpose of specific surface area of  $33\text{m}^2/\text{g}$  was obtained.

[0014] In example 3 example 1, except using 10.7g ( $\text{PCl}_3$ ) of phosphorus trichlorides, it processed similarly and the conductive impalpable powder (sample C) made into the purpose of specific surface area of  $31\text{m}^2/\text{g}$  was obtained.

[0015] In example 4 example 2, except maintaining Neutralization pH to 9.0-9.5, it processed similarly and the conductive impalpable powder (sample D) made into the purpose of specific surface area of  $39\text{m}^2/\text{g}$  was obtained.

[0016] Concurrency addition of the solution which dissolved 500g ( $\text{SnCl}_4$  and  $5\text{H}_2\text{O}$ ) of stannic chlorides in 500ml of 5-N hydrochloric acids, the solution which dissolved 7.4g ( $\text{H}_3\text{PO}_4$ ) of phosphoric acids in 200ml of water, and the 3-N sodium-hydroxide water solution was carried out over for 30 minutes, maintaining pH of a system to 6.0-7.0, and precipitate was made to generate in 5l. of water of 560 degrees C of examples. Next, after adding the hydrochloric acid and adjusting pH of a system to 3.0, this precipitate was filtered, and it washed until the specific resistance of filtrate was set to 10000-ohmcm after that. After drying the obtained cake at 110 degrees C for 12 hours, it calcinated at 1000 degrees C with the electric furnace for 1 hour, the PARUPE riser ground, and the conductive impalpable powder (sample E) made into the purpose of specific surface area of  $32\text{m}^2/\text{g}$  was obtained.

[0017] Concurrency addition of the solution which dissolved 500g ( $\text{SnCl}_4$  and  $5\text{H}_2\text{O}$ ) of stannic chlorides in 500ml of 5-N hydrochloric acids, and the solution which dissolved 10.8g ( $\text{Na}_2\text{HPO}_4$ ) of disodium hydrogenphosphate in 3-N sodium-hydroxide water solution was carried out over for 20 minutes, maintaining pH of a system to 6.0-7.0, and precipitate was made to generate in 5l. of water of 660 degrees C of examples. Next, after adding the hydrochloric acid and adjusting pH of a system to 3.0, this precipitate was filtered, and it washed until the specific resistance of filtrate was set to 10000-ohmcm after that. After drying the obtained cake at 110 degrees C for 12 hours, it calcinated at 1000 degrees C with the electric furnace for 1 hour, the PARUPE riser ground, and the conductive impalpable powder (sample F) made into the purpose of specific surface area of  $40\text{m}^2/\text{g}$  was obtained.

[0018] Concurrency addition of the solution and sodium-hydroxide water solution which dissolved 300g ( $\text{SnCl}_4$  and  $5\text{H}_2\text{O}$ ) of stannic chlorides in 900ml of 3-N hydrochloric acids was carried out for pH of a system over for 20 minutes, maintaining to 7.0-7.5, and precipitate was made to generate in 5l. of water of 750 degrees C of examples. Subsequently, the hydrochloric-acid water solution in which 8.05g ( $\text{PCl}_3$ ) of phosphorus trichlorides was dissolved was added to this precipitate, pH of a system was adjusted and stirred to 3, and the Lynn content water tin-oxide precipitate was obtained. This precipitate was filtered, and it washed until the specific resistance

of filtrate became subsequently to 10000-ohmcm. After drying the obtained cake at 110 degrees C for 12 hours, it calcinated at 1000 degrees C with the electric furnace for 1 hour, the PARUPE riser ground, and the conductive impalpable powder (sample G) made into the purpose of specific surface area of  $34.5 \text{ m}^2 / \text{g}$  was obtained.

[0019] Concurrency addition of the solution and sodium-hydroxide water solution which dissolved 300g ( $\text{SnCl}_4$  and  $5\text{H}_2\text{O}$ ) of stannic chlorides and 4.41g ( $\text{H}_3\text{PO}_4$ ) of orthophosphoric acid in 300ml of 3-N hydrochloric acids was carried out for pH of a system over for 20 minutes, maintaining to 7.0-7.5, and precipitate was made to generate in 5l. of water of 890 degrees C of examples. Next, after adding the hydrochloric-acid water solution and adjusting pH of a system to 2.5, this precipitate was filtered, and it washed until the specific resistance of filtrate became subsequently to 10000-ohmcm. After drying the obtained cake at 110 degrees C for 12 hours, it calcinated at 1150 degrees C with the electric furnace for 2 hours, the PARUPE riser ground, and the conductive impalpable powder (sample H) made into the purpose of specific surface area of  $14.5 \text{ m}^2 / \text{g}$  was obtained.

[0020] In example 9 example 8, except making burning temperature into 1100 degrees C, it processed similarly and the conductive impalpable powder (sample I) made into the purpose of specific surface area of  $21.1 \text{ m}^2 / \text{g}$  was obtained.

[0021] In example of comparison 1 example 1, except not using a phosphorus trichloride ( $\text{PCl}_3$ ), it processed similarly and the powder (sample J) of specific surface area of  $8 \text{ m}^2 / \text{g}$  was obtained.

[0022] In example of comparison 2 example 1, except using 4.9g ( $\text{PCl}_3$ ) of phosphorus trichlorides, it processed similarly and the impalpable powder (sample K) of specific surface area of  $32 \text{ m}^2 / \text{g}$  was obtained.

[0023] In example of comparison 3 example 1, except using 31.3g ( $\text{PCl}_3$ ) of phosphorus trichlorides, it processed similarly and the impalpable powder (sample L) of specific surface area of  $32 \text{ m}^2 / \text{g}$  was obtained.

[0024] Maintaining the solution and 3-N sodium-hydroxide water solution which dissolved 500g ( $\text{SnCl}_4$  and  $5\text{H}_2\text{O}$ ) of stannic chlorides in 500ml of 5-N hydrochloric acids to pH 6.0-7.0 of a system, concurrency addition was carried out over for 20 minutes, and precipitate was made to generate in 5l. of pure water of 450 degrees C of examples of a comparison. Next, after adding the hydrochloric acid and adjusting pH of a system to 3.0, this precipitate was filtered, and it washed until the specific resistance of filtrate was set to 10000-ohmcm after that. Using the inert-gas-replacement mold electric furnace, baking of 2 hours was performed at 350 degrees C among the nitrogen gas air current, the PARUPE riser ground the obtained cake, and the impalpable powder (sample M) of specific surface area of  $27 \text{ m}^2 / \text{g}$  was obtained.

[0025] In the example 1 of example of comparison 5 comparison, except making burning temperature into 600 degrees C, it processed similarly and the impalpable powder (sample N) of specific surface area of  $32 \text{ m}^2 / \text{g}$  was obtained.

[0026] In example of comparison 6 example 2, except making burning temperature into 600 degrees C, it processed similarly and the impalpable powder (sample O) of specific surface area of  $83 \text{ m}^2 / \text{g}$  was obtained.

[0027]

[Test Example(s)] The fine-particles resistance, specific surface area, and stability with the passage of time were measured by the following approaches about the sample powder obtained in the above, the example, and the example of a comparison.

[0028] (1) It is the evaluation sample powder of fine-particles resistance  $100 \text{ kg/cm}^2$  It fabricated by the pressure, and considered as the cylindrical green compact (3mm in the diameter of 18mm, thickness), the direct current resistance was measured, and it asked for fine-particles resistance ( $\omega \text{ gcm}$ ) from the following formula.

[Fine-particles resistance] = [measured-value] x [2.54-/thickness]

(For 2.54 in a formula, the unit of an electrode constant and thickness is cm)

[0029] (2) The test portions 0.1-0.2g of specific surface area were extracted, and it deaerated in nitrogen gas for 30 minutes at 150 degrees C. Then, specific surface area was measured with the BET adsorption method by nitrogen / helium mixed-gas system using the specific-surface-area measuring device (flow SOBU2300 mold: microphone ROMERI tick company make).

[0030] (3) After extending 10g of evaluation samples of the stability of fine-particles resistance with the passage of time on the filter paper and leaving it for one week at a room temperature, fine-particles resistance was measured. These results are shown in Table 1. (M of front Naka shows a megger and K shows kilo.)

[0031]

[Table 1]

試料		処理条件			特性		
		処理剤	処理量	焼成温度 (°C)	比表面積 (m <sup>2</sup> /g)	粉体抵抗 (Ω・cm)	粉体抵抗 (Ω・cm 一週間後)
		リン化合物	P/Sn (原子比)				
A	実施例 1	PCl <sub>3</sub>	$8.2 \times 10^{-2}$	1000	3.4	7.5	7.0
B	" 2	"	$6.8 \times 10^{-2}$	"	3.3	2.5	2.7
C	" 3	"	$5.5 \times 10^{-2}$	"	3.1	1.08	9.8
D	" 4	"	$6.8 \times 10^{-2}$	"	3.9	1.5	1.5
E	" 5	H <sub>3</sub> PO <sub>4</sub>	$5.3 \times 10^{-2}$	"	3.2	1.1	1.3
F	" 6	Na <sub>2</sub> HPO <sub>4</sub>	$5.3 \times 10^{-2}$	"	4.0	2.1	2.0
G	" 7	PCl <sub>3</sub>	$6.8 \times 10^{-2}$	"	3.4.5	1.60	1.56
H	" 8	H <sub>3</sub> PO <sub>4</sub>	$5.3 \times 10^{-2}$	1150	1.4.5	1.6	1.5
I	" 9	H <sub>3</sub> PO <sub>4</sub>	$5.3 \times 10^{-2}$	1100	2.1.1	1.2	1.2
J	比較例 1	—————	—————	1000	8	1.4M	1.4M
K	" 2	PCl <sub>3</sub>	$2.5 \times 10^{-2}$	"	3.2	2.3M	2.4M
L	" 3	"	$1.6 \times 10^{-1}$	"	3.2	1.2M	1.2M
M	" 4	—————	—————	350	2.7	8.5	1.0K
N	" 5	—————	—————	600	3.2	1.8M	1.8M
O	" 6	PCl <sub>3</sub>	$6.8 \times 10^{-2}$	"	8.3	4.5K	4.5K

[0032]

[Effect of the Invention] The conductive impalpable powder of the invention in this application is white impalpable powder excellent in conductivity which Lynn of the amount of specification fully dissolves during a tin-oxide crystal, is excellent in safety, and does not have the feeling of a blueness color tone which is moreover seen in antimony use. The conductivity of the conductive impalpable powder of the invention in this application is stabilized with time, and is useful as fillers, such as a coating aiming at electrification prevention, ink, plastics, rubber, and fiber, as electro-conductivity applying agents, such as electrophotography sensitized paper and electrostatic recording paper. Moreover, in order to manufacture the conductive impalpable powder of the invention in this application, 800-1300 degrees C in order to calcinate by high temperature comparatively, the Lynn component dissolves enough in a tin-oxide crystal, moreover growth of a particle and sintering do not arise, but specific surface area can manufacture industrially the ultrafine particle 10m<sup>2</sup> / more than g.

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CLAIMS

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[Claim(s)]

[Claim 1] Conductive impalpable powder whose specific surface area the tin oxide is used as a principal component, Lynn is included by the P/Sn atomic ratio at a rate of  $2.7 \times 10^{-2}$  to  $1.4 \times 10^{-1}$ , and is more than  $10 \text{ m}^2 / \text{g}$  and whose fine-particles resistance is below 500-ohmcm.

[Claim 2] Conductive impalpable powder according to claim 1 which includes Lynn by the P/Sn atomic ratio at a rate of  $5.0 \times 10^{-2}$  to  $9.0 \times 10^{-2}$ .

[Claim 3] It is made the water tin-oxide precipitate which make a fusibility tin compound solution react to the bottom of existence of (1) fusibility phosphorus-compounds solution with an acid or alkali, or it is made to react with (2) acids or alkali, and generates it whether addition processing of the fusibility phosphorus compounds is carried out. Obtain the Lynn content water tin-oxide precipitate, and after an appropriate time, classify the obtained precipitate, and it calcinates and grinds at the temperature of 800-1300 degrees C. How to manufacture the conductive impalpable powder whose specific surface area use the tin oxide as a principal component, include Lynn by the P/Sn atomic ratio at a rate of  $2.7 \times 10^{-2}$  to  $1.4 \times 10^{-1}$ , and is more than  $10 \text{ m}^2 / \text{g}$  and whose fine-particles resistance is below 500-ohmcm.

[Claim 4] The coating constituent characterized by including conductive impalpable powder according to claim 1 or 2 ten to 80% of the weight.

[Claim 5] The water or the organic solvent dispersing element characterized by including conductive impalpable powder according to claim 1 or 2 ten to 80% of the weight.

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